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HEAT RESISTANT EXPLOSIVES XVIII.  
RECRYSTALLIZATION TECHNIQUES  
FOR DIPAM (C)

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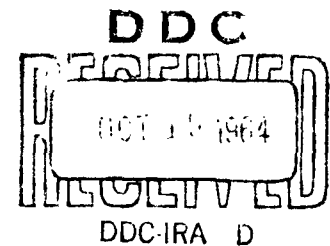
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HEAT RESISTANT EXPLOSIVES XVIII.  
RECRYSTALLIZATION TECHNIQUES FOR DIPAM (C)

by

R. E. Oesterling and J. C. Dacons

ABSTRACT: The recrystallization of 3,3'-diamino-2,2',4,4',6,6'-hexanitrobiphenyl, DIPAM, from a series of two-solvent systems has been studied. Mixtures of tetrahydrofuran-toluene and tetrahydrofuran-carbon tetrachloride have given the best results. Using standard, as well as special continuous extraction techniques, the bulk density of crystalline DIPAM may be controlled over a wide range from 0.26 g/ml to 0.77 g/ml.

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9 July 1964

The production of DIPAM and its use in high temperature mild detonating fuse and other explosive components for missiles, aircraft and spacecraft has been described in NOLTR's 62-82, 62-175, 63-16, 63-285, and 64-94. This report describes recrystallization techniques for control of the bulk density of pure DIPAM. The work was performed under task PR-3.

R. E. ODENING  
Captain, USN  
Commander

*Albert Lightbody*  
ALBERT LIGHTBODY  
By direction

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## INTRODUCTION

DIPAM has been observed to give only a monoclinic crystal form with angles of  $90^\circ$ ,  $90^\circ$ , and  $95^\circ 3'$ . (1). Because of a strong tendency of DIPAM to supersaturate and its unfavorable solubility/temperature relation in one-solvent systems, the original recrystallization process was devised using acetone-ethanol. Crude DIPAM was dissolved in acetone at about 2.6g/100 ml, treated with decolorizing charcoal, then concentrated by boiling during the addition of absolute ethanol (2). This procedure, costly in both time and materials, invariably gives a small particle size product in the range of 10 to 30 microns, Fig. 1. The bulk density of DIPAM prepared by this process ranges from 0.26 to 0.35 g/ml; the more rapid concentration and addition of ethanol giving smaller particle size and lower bulk density.

Both particle size and shape control bulk density, which is a convenient measure of the net effect of these two variables. In some applications of DIPAM, bulk density may be a critical factor. Problems have been reported in the loading of DIPAM into small diameter tubing for the manufacture of mild detonating fuse. Material of fine particle size and low bulk density has poor flow properties, may create a serious dusting problem and causes difficulty in achieving maximum loading density.

This study of the recrystallization of DIPAM from a series of solvents and solvent pairs was undertaken for the purpose of controlling bulk density and this has been found to be feasible over a wide range from 0.26 to 0.77 g/ml.

## DISCUSSION AND RESULTS

Solvents and Solvent Pairs. The pure solvents tried were, in order of increasing solubility of DIPAM, acetone, tetrahydrofuran, dioxane, nitrobenzene and dimethyl formamide. None of these alone was found to give satisfactory results. In most cases the crystals were fine needles or thin plates, Fig. 2, the recovery was low, and in the case of the high boiling solvents a detrimental effect was noted on the vacuum stability of the product. The American Cyanamid Company (3) has reported the use of ethylene glycol as a recrystallizing solvent to give DIPAM of bulk density 0.46 g/ml. Problems were also encountered here in the vacuum stability and in scaling the process to larger samples.

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This study has shown that DIPAM may be recrystallized from the following list of solvent pairs:

tetrahydrofuran-toluene  
tetrahydrofuran-carbon tetrachloride  
tetrahydrofuran-xylene  
tetrahydrofuran-nitrobenzene  
acetone-acetic acid  
acetone-carbon tetrachloride  
dioxane-toluene  
dioxane-xylene

Some of these solvent pairs give DIPAM with relatively equal rates of growth of the crystal faces; thus under controlled conditions large thick prisms and near equants may be formed with bulk densities as high as 0.77 g/ml.

Preferred Recrystallization for Production of Pure DIPAM (4).  
The solubility of DIPAM in boiling tetrahydrofuran is approximately 10 g/100 ml. Such a solution of crude DIPAM may be conveniently treated with Darco G-60 and filtered. The tetrahydrofuran is then distilled off at a slow steady rate and toluene is added to the solution at approximately the same rate until crystallization is complete. During recrystallization the solution is stirred slowly to prevent bumping. After cooling, the DIPAM is filtered, washed with methanol and dried to give 94% recovery of material which pours well, with a bulk density of 0.38 g/ml, Fig. 3, and has a melting point of 305°C (dec). When wet sieved with n-hexane using 210, 105 and 44 micron sieves, 50% was caught on the 105, 40% on the 44 and 10% passed through. The recommended process for the production of DIPAM requires both tetrahydrofuran and toluene (4); thus the recovery of tetrahydrofuran by refractionation of the production as well as the recrystallizing solvents should decrease costs significantly.

Continuous Extraction-Recrystallization of DIPAM for High Bulk Density. Some factors which tend to give small particle size, low bulk density crystals are a high degree of supersaturation, spontaneous nucleation and a rapid rate of crystallization (5). These factors may be controlled to a large extent by the use of continuous extraction techniques. By the choice of a suitable ratio of a low boiling good solvent mixed with a higher boiling poor solvent in an ordinary laboratory Soxhlet extractor, the rate of feed of saturated solution to the recrystallizing flask may be adjusted to minimize supersaturation. With crude DIPAM in the extractor thimble, the recrystallizing flask is seeded with DIPAM crystals to avoid spontaneous nucleation, and stirred

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to equalize the diffusion effects and help to give more equal rates of growth of the crystal faces.

A continuous extraction-recrystallization apparatus with a fritted glass disc, Fig. 4, was also used successfully for producing high bulk density DIPAM. This apparatus with a solvent mixture of tetrahydrofuran-toluene, 1:2, operating at a moderate reflux rate for 20 hours produced DIPAM as thick prisms 100 to 200 microns long with a bulk density of 0.50g/ml, Fig. 5. With acetone-acetic acid, 1:1, flat plates of approximately 100 microns were obtained. The bulk density was 0.48g/ml, Fig. 6. Tetrahydrofuran-carbon tetrachloride, 2:1, gave relatively small crystals, 30 to 50 microns, of nearly equant shape with a bulk density of 0.55 g/ml, Fig. 7.

The effects of seeding of the recrystallizing flask were most apparent in the experiments giving the highest bulk density crystals. When the tetrahydrofuran-carbon tetrachloride, 2:1, system was seeded with material of 0.55 g/ml and operated for ten hours, the near cubic crystals had grown to about 100 microns with a bulk density of 0.65 g/ml, Fig. 8. Similarly, when the 0.65 g/ml material was used as seed in a solvent mixture of tetrahydrofuran-carbon tetrachloride, 1:1, after 20 hours operation the product bulk density was 0.77 g/ml, Fig. 9. It was also noted that in the seeded recrystallization experiments, if the reflux rate or extraction rate were too fast, spontaneous nucleation could occur and sufficient smaller crystals were formed to reduce the final bulk density.

The Soxhlet extraction apparatus because of its siphoning process delivers a less concentrated solution to the flask and is therefore a slower operation. In 20 hours the Soxhlet apparatus gave material of 0.66 g/ml, Fig. 10, using tetrahydrofuran-nitrobenzene, 1:1. With tetrahydrofuran-toluene, 1:3, without seeding, large thick prisms were formed with a bulk density of 0.71 g/ml, Fig. 11.

The disadvantage of the continuous extraction-recrystallization process is the time requirement. This is partially offset by the smaller volumes of solvents required. After 20 hours operation some of the solvent mixtures became dark in color, but these could be decolorized with Darco G-60 after filtration of the DIPAM and reused.

All the samples of crystalline DIPAM produced in these experiments had melting points of 304°C (dec) or higher, and vacuum stabilities at 260°C were within the limits of the present specifications. Microscopic examination of the crystals

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from tetrahydrofuran-toluene and tetrahydrofuran-carbon tetrachloride indicated these to be quite free of solvent occlusions.

ACKNOWLEDGEMENT

The authors wish to thank Dr. Jerome M. Rosen for the microscopic examination of the DIPAM crystals, and for preparing the photomicrographs reproduced here.

EXPERIMENTAL

Recrystallization of DIPAM from Tetrahydrofuran-Toluene.  
Combined miscellaneous samples, 287 g, of DIPAM, m. p.  $303^{\circ}\text{C}$  (dec) was dissolved in 3000 ml of boiling tetrahydrofuran\*. This bright yellow solution did not require treatment with charcoal; however, some crude DIPAM samples may give a dark colored solution and may be treated with 25-30 g of decolorizing charcoal, Darco G-60 per liter without appreciable losses. The solution was placed in a five-liter three-neck flask fitted with a stirrer, dropping funnel and a distillation take-off. The solution was stirred slowly and distilled at a moderate rate until 500 ml of distillate had been removed; then 1000 ml of toluene was added through the funnel at a rate sufficient to keep the volume approximately constant in the flask. Recrystallization began during this addition and stirring was adjusted to prevent bumping. When a total of 2000 ml of distillate had been removed the distillation temperature was  $100^{\circ}\text{C}$  and heating was discontinued. The mixture was allowed to cool for only a few minutes then filtered while still warm. The DIPAM was washed thoroughly with methanol then dried in a forced air oven at  $140\text{--}150^{\circ}\text{C}$  overnight. The recovery was 270 g, 94%, of bright yellow DIPAM, m. p.  $305^{\circ}\text{C}$  (dec); bulk density, 0.38 g/ml; vacuum stability, 20 min. surge = 0.65 cc, two hour period = 1.26 cc/g/hr. When a 5 g sample was wet sieved with n-hexane through 210, 105, and 44 micron sieves, 2.5 g was caught on the 105, 2.0 g on the 44 and 0.5 g passed through the 44 micron sieve.

---

\* "Baker Analyzed" Reagent Grade tetrahydrofuran containing 0.025% butylated hydroxytoluene as a stabilizer was used as received. All other solvents were also "Reagent Grade" and used as received.

Continuous Extraction-Recrystallization of DIPAM. The extraction thimble, 43 mm diameter, of a large Soxhlet apparatus was filled with 50 g of crude DIPAM. In the 1000 ml boiling flask with a Teflon covered magnetic stirring bar and a Glas-Col heating mantle was placed 800 ml of the solvent mixture. The solvent was stirred at about 200 rpm and heated to give a reflux rate causing siphoning every 4-5 min. The thimble could be removed, refilled and replaced without interruption of the reflux rate. At the end of the 10-20 hour recrystallization period, reflux was discontinued and the contents of the flask filtered hot. The crystalline DIPAM was washed several times with methanol then dried at 150°C in a forced air oven for 15 hours. The results of typical experiments are listed in Table 1.

A charge of crude DIPAM, 200 g, was placed in the body of the fritted glass disc extraction apparatus, Fig. 4, with 800 ml of the solvent mixture in the 1000 ml boiling flask stirred and heated as above. For the seeded experiments 15 to 25 g of recrystallized DIPAM of measured bulk density was added to the solvent before reflux was started. The apparatus was fitted with a water cooled sealed stirrer and a reflux condenser. For solvent mixtures of tetrahydrofuran (THF) - toluene the boiling rate was controlled to avoid build-up of a liquid layer in the body of the extractor. For solvent mixtures of slower extraction rates such as the THF-CCl<sub>4</sub> the reflux rate was increased to maintain a stirrable slurry of solvent and DIPAM. The crystals were filtered, washed and dried as above. Results are listed in Table 1. The filtrates, except in the case of the THF-nitrobenzene, were treated with 20 g of Darco-G-60 for reuse. The color of fine particle size DIPAM is a bright yellow; however, as the particle size and bulk density increased the color changed to orange and these colors were not changed during the drying process.

Bulk Density Measurement. Dry DIPAM powder, 10.0 grams, was introduced into a clean, dry 50 ml graduated cylinder through a 60° Pyrex glass funnel of 65 mm top diameter with a one inch stem of about 11 mm I.D. For samples of low bulk density slow sifting through the funnel was necessary to avoid plugging or hold-up. With the cylinder held securely on the bench-top, it was tapped lightly five times on the side with a wooden pencil to level the powder surface. The volume was read to the nearest milliliter and divided into the sample weight to give the bulk density in grams/ml.

TABLE 1

## CONTINUOUS EXTRACTION-RECRYSTALLIZATION OF DIPAM

Solvent Mixture	Ratio	Seeding	Operating Time (hrs)	Recrystallized Product Weight(g)	Bulk Density	Fig. #
THF-Toluene	1:2	20g of 0.38g/ml	20	210	0.50 g/ml	5
Acetone-Acetic acid	1:1	none	18	180	0.48	6
THF-CCl <sub>4</sub>	2:1	none	14	185	0.55	7
THF-CCl <sub>4</sub>	2:1	25g of 0.55g/ml	10	150	0.65	8
THF-CCl <sub>4</sub>	1:1	15g of 0.65g/ml	20	100	0.77	9
SOXHLET EXTRACTOR						
THF-Nitrobenzene	1:1	none	20	60	0.66	10
THF-Toluene	1:3	none	20	80	0.71	11


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100  $\mu$   
DIPAM — 50X  
RECRYSTALLIZED FROM ACETONE-ETHANOL  
BULK DENSITY = 0.26 G/ML

FIG. 1



100  $\mu$   
DIPAM — 50X  
RECRYSTALLIZED FROM NITROBENZENE

FIG. 2

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100 $\mu$   
DIPAM     $\equiv$     50X  
RECRYSTALLIZED FROM TETRAHYDROFURAN-TOLUENE  
BULK DENSITY = 0.38 G/ML

FIG. 3

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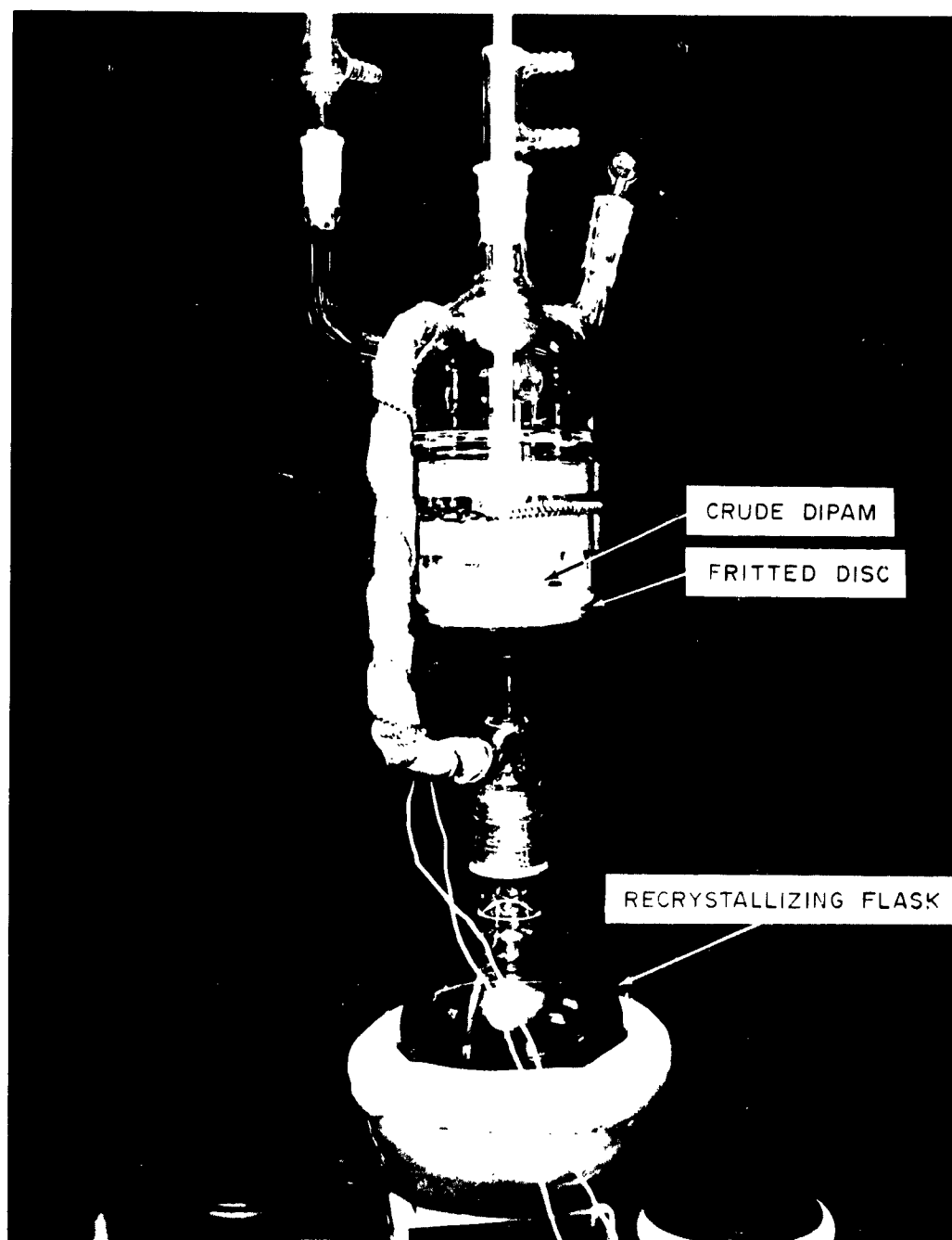


FIG. 4 CONTINUOUS EXTRACTION - RECRYSTALLIZATION APPARATUS  
DESIGNED BY L. A. KAPLAN

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100  $\mu$   
DIPAM  $\text{H}$  50X

CONTINUOUS EXTRACTION WITH TETRAHYDROFURAN - TOLUENE (1:2)  
BULK DENSITY = 0.50 G/ML

FIG. 5



100  $\mu$   
DIPAM  $\text{H}$  50X


CONTINUOUS EXTRACTION WITH ACETONE - ACETIC ACID  
BULK DENSITY = 0.48 G/ML

FIG. 6

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100 $\mu$   
DIPAM  50X

CONTINUOUS EXTRACTION WITH TETRAHYDROFURAN- $\text{CCl}_4$  (2:1)  
BULK DENSITY = 0.55 G/ML

FIG. 7



100 $\mu$   
DIPAM  50X

CONTINUOUS EXTRACTION WITH TETRAHYDROFURAN- $\text{CCl}_4$  (2:1)  
BULK DENSITY = 0.65 G/ML

FIG. 8

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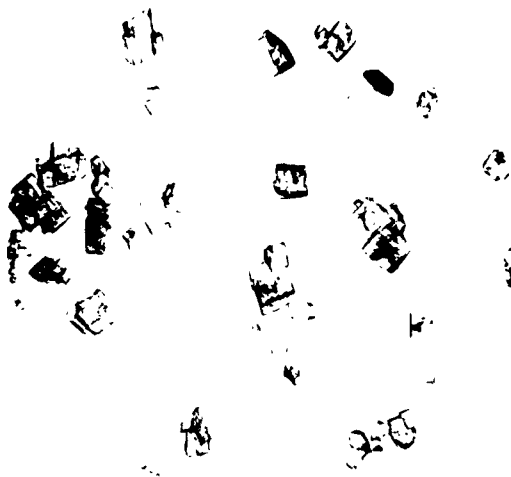
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DIPAM  $100\mu$   
H 50X

CONTINUOUS EXTRACTION WITH TETRAHYDROFURAN- $\text{CCl}_4$  (1:1)  
BULK DENSITY = 0.77 G/ML

FIG. 9



DIPAM  $100\mu$   
H 50X

SOXHLET EXTRACTION WITH TETRAHYDROFURAN-NITROBENZENE (1:1)  
BULK DENSITY = 0.66 G/ML

FIG. 10

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100 $\mu$   
DIPAM     $\text{H}$     50X  
SOXHLET EXTRACTION WITH TETRAHYDROFURAN-TOLUENE (1:3)  
BULK DENSITY = 0.71 G/ML

FIG. 11

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Mild		MILD	Amino		AMIN			
Detonating		DETO	Exploding		EXPT			

<p>1. Naval Ordnance Laboratory, White Oak, Md. (NOL technical report 64-102) HEAT RESISTANT EXPLOSIVES XVIII. RECRYSTALLIZATION TECHNIQUES FOR DIPAM (C), by R. E. Oesterling and J. C. Dacons. 9 July 1964. 7p. illus., tables. NOL task PR-3.</p> <p>CONFIDENTIAL</p> <p>This report describes the recrystallization of 3,3'-diamino-2,2',4,4',6,6'-hexanitrophenyl, DIPAM, from a series of two-solvent systems. Mixing tetrahydrofuran-toluene and tetrahydrofuran-carbon tetrachloride give the best results. Using standard and special continuous extraction techniques the bulk density of crystalline DIPAM may be controlled over a wide range from 0.26 to 0.77 g/ml.</p> <p>DOWNGRADED AT 3 YEAR INTERVALS DECLASSIFIED AFTER 12 YEARS DOD DIR 5200.10.</p>	<p>1. 3,3'-Diamino-2,2',4,4',6,6'-hexanitro-biphenyl Explosives I. Title II. Oesterling, Robert E. III. Dacons, Joseph C., jt. author IV. Project V. Title: Recrystallization...(U).</p> <p>Abstract card is confidential.</p>	<p>1. 3,3'-Diamino-2,2',4,4',6,6'-hexanitro-biphenyl Explosives I. Title II. Oesterling, Robert E. III. Dacons, Joseph C., jt. author IV. Project V. Title: Recrystallization...(U).</p> <p>Abstract card is confidential.</p>
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